

Relaxation measurements in the regime of the second magnetization peak in Nb films

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We report on magnetic measurements as a function of field, temperature and time (relaxation) in superconducting Nb films of critical temperature $T_c = 9.25$ K. The magnetic measurements as a function of field exhibited a "second magnetization peak" ("SMP") which in general is accompanied by thermomagnetic instabilities (TMIs). The lines $H''_{\text{smp}}(T)$ where the "SMP" occurs and the $H_{\text{fj}}(T)$ where the first flux jump in the virgin magnetization curves is observed, end at a characteristic point $(T_o, H_o) \approx (7.2 \text{ K}, 80 \text{ Oe})$. Relaxation measurements showed that for $T < T_o \approx 7.2$ K the activation energy U_o and the normalized relaxation rate S exhibit non-monotonic behavior as a function either of temperature or field. The extrema observed in U_o and S coincide with the points $H_{\text{on}}(T)$ or $H''_{\text{smp}}(T)$ of the "SMP". In the regime $T > T_o \approx 7.2$ K both U_o and S present a conventional monotonic behavior. These results indicate that the "SMP" behavior observed in our Nb films is promoted by the anomalous relaxation of the magnetization.

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INTRODUCTION

Recent magnetic studies in Nb films [1, 2, 3] reported the existence of a structure that reminisces of the second magnetization peak (SMP) which is usually observed in high- T_c superconductors [4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19]. These studies concluded that for the case of Nb films the "SMP" is probably motivated by thermomagnetic instabilities (TMIs) that occur in the low-temperature regime, far below the upper-critical field [1, 2, 3]. From the theoretical point of view, the understanding of the fundamental differences between the "SMP" observed in Nb films and in high- T_c superconductors is of great importance. In addition, the detailed experimental study of the TMIs that accompany the "SMP" in Nb films is also very important, because these undesirable flux jumps constitute a serious limitation for basic practical applications.

Today, a detailed study of the dynamic response of vortices in the regime of the "SMP" in Nb films is lacking. In this work we present detailed magnetic data and especially we study the relaxation of vortices in sputtered thick Nb films that exhibit the "SMP". We systematically recorded the lines $H''_{\text{smp}}(T)$ where the "SMP" occurs, $H_{\text{fj}}(T)$ where the first flux jump takes place in the virgin magnetization curves and $H_{\text{fp}}(T)$ where the first peak is observed. These three lines connect at a characteristic point $(T_o, H_o) \approx (7.2 \text{ K}, 80 \text{ Oe})$. For $T > T_o$ no flux jumps are observed and the magnetic curves are absolutely smooth. Interestingly, below a second characteristic point $(T_1, H_1) \approx (6.2 \text{ K}, 1600 \text{ Oe})$ the line $H''_{\text{smp}}(T)$ changes slope. In the regime $T_1 < T < T_o$

TMIs are pronounced and the "SMP" fades as the characteristic temperature T_o is approached. In contrast, for $T < T_1$ TMIs are strongly suppressed. As a consequence, in this regime the magnetic curves are almost regular and the "SMP" strongly resembles the respective feature observed in high- T_c compounds. Detailed relaxation measurements, showed that the activation energy U_o (normalized relaxation rate S) exhibits a maximum (minimum) as a function of temperature or field for $T < T_o \approx 7.2$ K. The observed extrema are located exactly at the $H''_{\text{smp}}(T)$ or $H_{\text{on}}(T)$ points. In contrast, in the regime $T > T_o \approx 7.2$ K a monotonic behavior is observed for both U_o and S . Although our results indicate that in Nb films the "SMP" feature is simply motivated by an anomalous relaxation mechanism, the similarities of the observed behavior with the behavior observed in high- T_c compounds calls for further theoretical and experimental work.

PREPARATION OF THE FILMS AND EXPERIMENTAL DETAILS

The samples of Nb were sputtered on Si [001] substrates as they were annealed during the deposition. Details on the preparation of the films are presented elsewhere [3]. The critical temperature of the film under discussion is $T_c = 9.25$ K, equal to the one of high purity single crystals [20]. The residual resistance ratio is $R(300 \text{ K})/R(10 \text{ K})=6.8$ ($\rho_n \approx 10 \mu\Omega\text{cm}$ just above T_c). The thickness of the film is 7700 \AA . Our combined x-ray diffraction (XRD) and transmission electron microscopy (TEM) data revealed that by annealing the films during deposition, a larger mean size of the grains is produced, which in this case is 930 \AA . Furthermore, the grains of the annealed films are oriented, in some degree, with [110] direction perpendicular to the film's surface. Our magnetic measurements either as a function of field,

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temperature or time were performed by means of a commercial SQUID magnetometer (Quantum Design). In all magnetic data presented below, the magnetic field was always normal to the surface of the film ($\mathbf{H} \parallel \mathbf{c}$). All the magnetic measurements were performed under zero field cooling (ZFC), so that we always obtained the virgin magnetization curves. In relaxation measurements the magnetization of the sample was recorded for a time window of $100 \text{ sec} < t < 4200 \text{ sec}$. We examined the whole temperature-magnetic-field regime accessible by our SQUID ($H_{\text{dc}} < 55 \text{ kOe}$, $T > 1.8 \text{ K}$).

EXPERIMENTAL RESULTS AND DISCUSSION

TMIs and the "SMP" in magnetic measurements

We start the presentation of our experimental results with the magnetic measurements as a function of field. Figures 1(a) and 1(b) present data in the high (upper panel) and in the low-temperature regime (lower panel). We see that close to T_c (upper panel) the magnetic curves are smooth, while the loops at low temperatures (lower panel) are anomalous. More specifically, below the characteristic temperature $T_o = 7.2 \text{ K}$ a "noisy" first peak shows up which is accompanied by strong TMIs. As the temperature is gradually reduced this broad peak transforms into two separate peaks: a first maximum which occurs in low-field values, and a distinct second peak which is placed in high magnetic fields. We note that the magnetic loops performed in the interval $T_1 \approx 6.2 \text{ K} < T < T_o \approx 7.2 \text{ K}$ exhibited strong TMIs. In this regime the "SMP" is hardly observed. In contrast, for even lower temperatures $T < T_1 \approx 6.2 \text{ K}$, the "SMP" is pronounced and the TMIs are strongly suppressed. As a result the magnetic curves are more regular. The overall behavior resembles the SMP observed usually in high- T_c superconductors [4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19]. For this reason we "loosely" refer to these characteristic fields as H''_{smp} . However, there are noticeable differences between the magnetic behaviors observed in our Nb film and in high- T_c superconductors. In our case, below the "SMP" ($H < H''_{\text{smp}}$) the response is "noisy", while above it ($H > H''_{\text{smp}}$) we observed smooth magnetic curves. In contrast, in *point disordered* high- T_c superconductors the magnetization curves are smooth, both below and above the SMP. In addition, the peak value of the H''_{smp} slightly decreases as we lower the temperature. In high- T_c superconductors for lower temperatures the peak value of the SMP strongly increases. Finally, in our Nb film the resulting line $H''_{\text{smp}}(T)$ ends on the first peak line $H_{\text{fp}}(T)$ at $T_o/T_c \approx 0.78$ (see Fig. 3 below), while in high- T_c superconductors the respective line $H_{\text{smp}}(T)$ ends near the irreversibility/melting line, or extends almost up to the critical temperature. In the

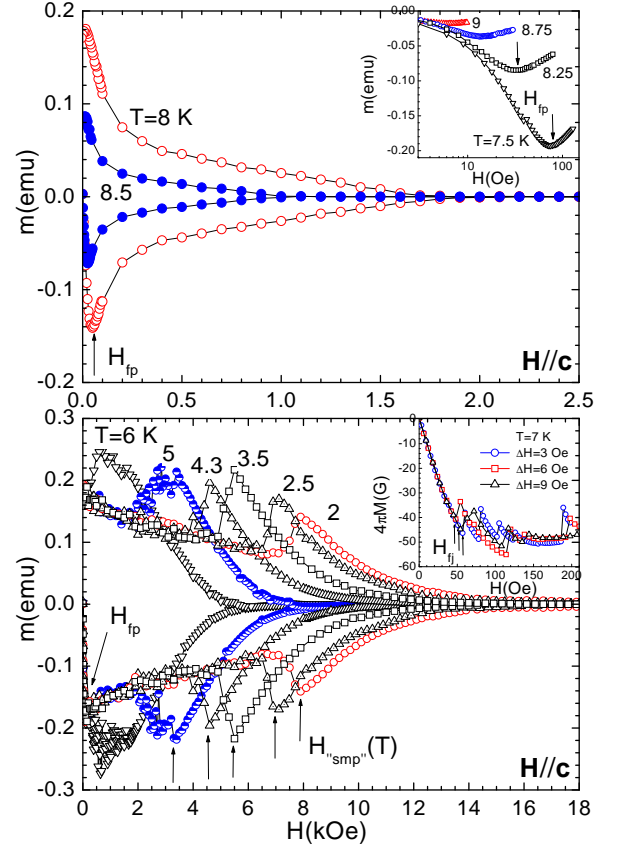


FIG. 1: Isothermal magnetic $m(H)$ measurements for $T = 8$ and 8.5 K , above $T_o \approx 7.2 \text{ K}$ (upper panel), and for $T = 6, 5, 4.3, 3.5, 2.5$ and 2 K , below $T_o \approx 7.2 \text{ K}$ (lower panel). For $T > T_o \approx 7.2 \text{ K}$ the magnetic curves are smooth, while for $T < T_o \approx 7.2 \text{ K}$ they are accompanied by TMIs, presenting a "SMP" feature at points H''_{smp} . At very low temperatures the TMIs are strongly suppressed and the "SMP" is evident. In the inset of the upper panel we present magnetic measurements in low magnetic fields, at various temperatures, for the determination of the first peak field H_{fp} . In the inset of the lower panel we focus on the flux jumps that occurred at $T = 7 \text{ K}$, for the determination of the first flux jump field H_{fj} . Magnetic curves for various sweep rates of the applied field are presented. In all cases the magnetic field was normal to the surface of the film $\mathbf{H} \parallel \mathbf{c}$.

respective inset of the upper (lower) panel we present measurements at low magnetic fields, for the demonstration of the first peak field H_{fp} (first flux jump field H_{fj}).

In Figs. 2(a)-(c) we present magnetic data as a function of temperature in various magnetic fields. In Fig. 2(a) we present data at low applied fields of the order of the first critical field. In addition to the raw data we also present two more curves that resulted after multiplying the original curves (a) and (b) (referring to $H = 10$ and 20 Oe) by a factor of 3 and $3/2$ respectively. We clearly see that the behavior of the low temperature regime is the one expected in the Meissner state since the resulting

$H_{fj}(T)$ line as a simple boundary above which TMIs occur [3].

Relaxation measurements in the regime of the "SMP"

By measuring the magnetic moment as a function of time (relaxation measurements) new information may be gained about the pinning mechanism. Furthermore, additional information at (very short or long) times not accessible by a realistic experiment may be obtained, by extrapolating the measured curves. Of course, this is permitted under the assumption that the relaxation mechanism doesn't change. Our main aim was the study of the dynamic behavior of vortices in the regime of the "SMP". To this end, we performed detailed relaxation measurements at temperatures $T < T_o$. In addition, relaxation measurements were performed at temperatures $T > T_o$ where the "SMP" is not observed, in order to be compared with the respective ones at $T < T_o$. In Fig. 4 we show representative data at $T = 5$ K for various fields $H = 2 - 10$ kOe. In the upper (lower) panel we present results in the field regime above (below) the "SMP" (see Fig. 6(a) below). It is clear that the magnetic moment relaxes according to a logarithmic law, as this is evident by its linear variation on a semilogarithmic plot. The curves of the upper panel are smooth and their slope changes slowly as the upper-critical field is approached. In the lower panel, where the measurements were performed in the regime of TMIs (see Fig. 6(a) below), small flux jumps are observed. These jumps are restricted in short measuring times. Interestingly, the slope of the curves below and above each jump is almost the same. Nevertheless, in such cases, in the processing of the data we took into account only the part of the curves placed above the jump.

Since we study a low- T_c superconductor which exhibits logarithmic relaxation we used the classical Anderson-Kim model [25] for the estimation of the activation energy U_o and the respective normalized relaxation rate S . For Anderson-Kim flux creep the potential barrier that vortices experience is linear on the applied current according to the relation $U(j) = U_o(1 - j/j_c)$. It comes out that the bulk screening currents that exist in the superconductor due to the pinning of vortices, relax according to a logarithmic law. The relaxation of the magnetization of a thin disk under a perpendicular magnetic field has been also treated analytically in Ref. 26. In that work it was pointed out that in the regime of steady state relaxation (at times much higher than a characteristic transient time) the relaxing magnetization is independent of the initial conditions of the sweep rate of the applied field. The result obtained for the steady state relaxation

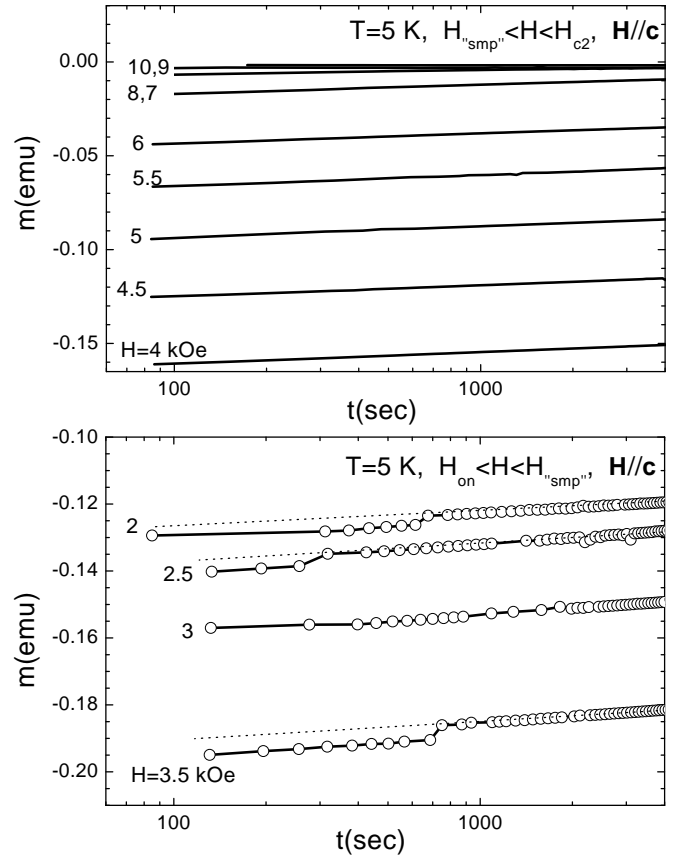


FIG. 4: Magnetic measurements $m(t)$ as a function of time under various magnetic fields $H = 2 - 10$ kOe, at $T = 5$ K. In the upper panel we present data in the field regime $H'_{smp''} < H < H_{c2}$, while in the lower panel we present respective data for $H_{on} < H < H'_{smp''}$. Clearly, the magnetic moment exhibits a logarithmic relaxation. In the lower panel we see that a few jumps are present, as these measurements were performed in the regime of TMIs (see Fig. 3 above). In all cases the magnetic field was normal to the surface of the film $\mathbf{H} \parallel \mathbf{c}$.

is

$$m(t) = m_c - m_1 \ln\left(\frac{t}{t_o}\right), \quad (1)$$

where $t_o \approx j_c da S / c^2 E_c$, $m_c = \pi a^3 j_c d / 3c$ and $m_1 = S m_c$. In the above quantities t_o is a characteristic microscopic attempt time, d and a are the thickness and the "diameter" of the sample respectively and finally, E_c is a threshold value of the electric field. By fitting the relaxation data we estimated the related pinning parameters U_o and S . In our fitting procedures we assumed that the attempt time is $t_o \in [10^{-3} - 10^{-6}]$ sec.

Figure 5 presents the estimated critical current density $j_c(H)$ at various temperatures. We clearly see that for $T < T_o \approx 7.2$ K $j_c(H)$ presents a non-monotonic behavior, while at $T > T_o \approx 7.2$ K a regular monotonic behavior is recovered. From these data we see that the "SMP"

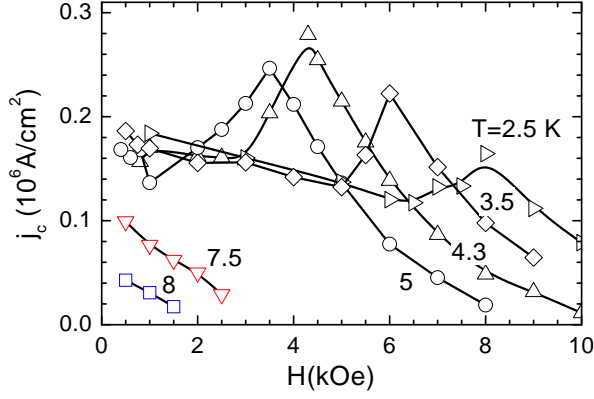


FIG. 5: Estimated critical current density at various temperatures $T = 2.5, 3.5, 4.3$ and 5 K (below $T_o \approx 7.2$ K) and at $T = 7.5$ and 8 K (above $T_o \approx 7.2$ K). For $T > T_o \approx 7.2$ K j_c is monotonic in the whole field regime, while for $T < T_o \approx 7.2$ K is non-monotonic presenting extrema as a function of field. In all cases the magnetic field was normal to the surface of the film $\mathbf{H} \parallel \mathbf{c}$.

feature is present even for the "unrelaxed" magnetization (at very small times of the order of $t_o \in [10^{-3} - 10^{-6}]$ sec).

In Fig. 6 we present comparatively the magnetic loops and the estimated activation energy U_o for various magnetic fields at $T = 5$ K (upper panel) and $T = 3.5$ K (lower panel). As we clearly see at very low magnetic fields, U_o takes very high values (consequently S takes very low values). When increasing the field, U_o exhibits a non-monotonic behavior. More specifically, for $T = 5$ K U_o presents a maximum at exactly the H''_{smp} point. At lower temperatures the maximum in U_o is placed in higher fields. For example, at $T = 3.5$ K the maximum in U_o is placed above the H''_{smp} point, while the local minimum of U_o is located at the onset point H_{on} of the "SMP". The appearance of these extrema in U_o (and also in S) means that there is an enhancement of the effective pinning in the regime of the "SMP". In a recent article H. Kupfer et al. [4] discussed relaxation as a possible origin of the SMP in $\text{YBa}_2\text{Cu}_3\text{O}_{7+\delta}$ single crystals. They proposed that the SMP could originate from a crossover between different relaxation mechanisms. Faster relaxation in the low field regime could lead to a suppression of the measured curves (that relax during the measurement), while slower relaxation at higher fields would restore higher values of the measured magnetization. As a consequence a SMP could be obtained. Under this point of view, if $j_c(H)$ was determined mainly by relaxation, a maximum of $j_c(H)$ should correspond to a minimum in S and a maximum in U_o . In the experiments of Ref. 4 the minimum in S (maximum in U_o) was observed at a characteristic field $H^*(T)$, placed well below the maximum point $H_{\text{smp}}(T)$ of the SMP. As a result the relaxation ori-

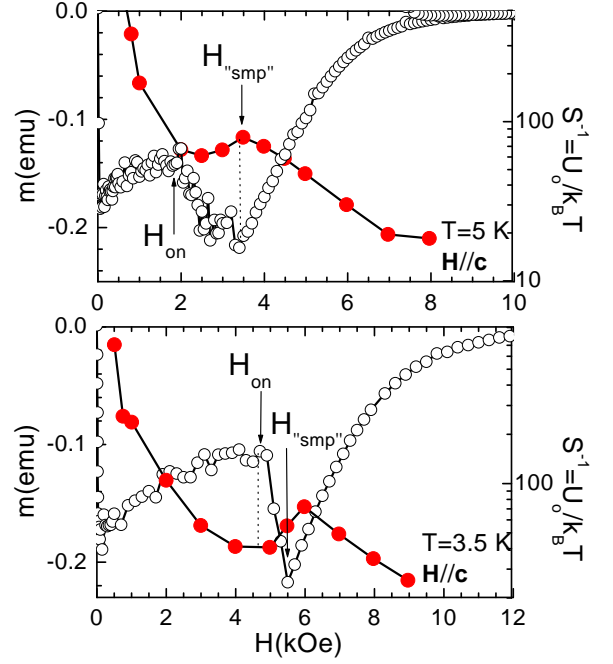


FIG. 6: The ascending branch of the magnetic measurement as a function of field and the estimated activation energy $U_o/k_B T$ in various magnetic fields, at $T = 5$ K (upper panel) and at $T = 3.5$ K (lower panel). We see that $U_o/k_B T$ presents a non-monotonic behavior. In intermediate temperatures ($T = 5$ K) the local maximum of $U_o/k_B T$ is located at exactly the peak point H''_{smp} of the "SMP". At lower temperatures ($T = 3.5$ K) it is the local minimum of $U_o/k_B T$ that coincides with the onset point H_{on} of the "SMP", while the local maximum of $U_o/k_B T$ is placed above the H''_{smp} point. In all cases the magnetic field was normal to the surface of the film $\mathbf{H} \parallel \mathbf{c}$.

gin of the SMP in $\text{YBa}_2\text{Cu}_3\text{O}_{7+\delta}$ single crystals remained a doubtful possibility. In our case, we see that both extrema of U_o are closely related to the onset $H_{\text{on}}(T)$ and the maximum $H''_{\text{smp}}(T)$ of the "SMP". This could indicate that in contrast to $\text{YBa}_2\text{Cu}_3\text{O}_{7+\delta}$, in our case the "SMP" feature could originate from an anomalous relaxation process as the magnetic field increases. Soon after, Y. Abulafia et al.[7] proposed that the anomalous behavior of S and U_o could be ascribed to a change in the dynamic response of vortices at $H^*(T) < H_{\text{smp}}(T)$. They proposed that for $H < H^*(T)$ the response of vortex system was elastic, while for $H > H^*(T)$ the increase of S (decrease of U_o) should be caused by plastic deformations of the vortex solid [7]. Since then, many experimental works confirmed this notion in $\text{YBa}_2\text{Cu}_3\text{O}_{7+\delta}$ and in other high- T_c compounds as $\text{HgBa}_2\text{CuO}_{4+\delta}$ and $\text{Tl}_2\text{Ba}_2\text{CaCu}_2\text{O}_8$ [4, 6, 10, 11, 18, 19]. More specifically, in high- T_c compounds for fields $H > H^*(T)$ the activation energy U_o follows a negative power-law $U_o \propto H^\nu$ with $\nu \in [-0.5, -0.9]$ [7, 10, 13, 18, 19]. In our case we observed that above the "SMP" the activation energy

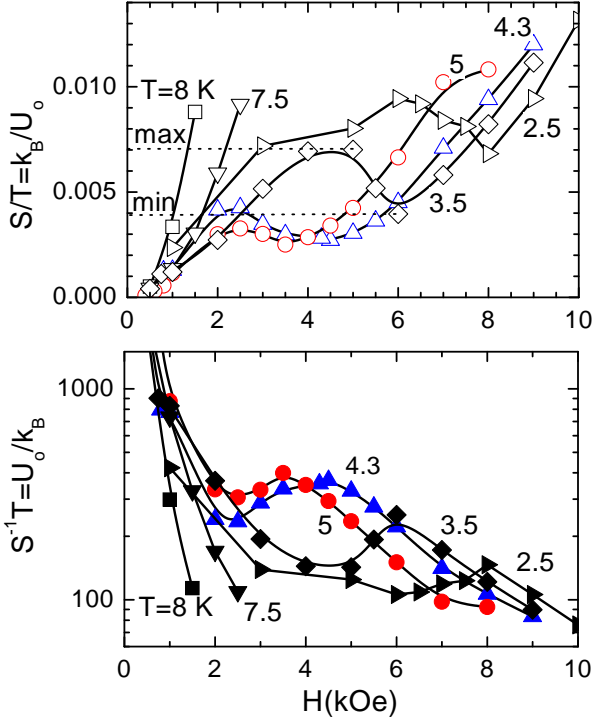


FIG. 7: Normalized relaxation rates S/T (upper panel) and activation energies U_o/k_B (lower panel) at temperatures $T = 2.5, 3.5, 4.3$ and 5 K (below $T_o \approx 7.2$ K) and at $T = 7.5$ and 8 K (above $T_o \approx 7.2$ K). For $T > T_o \approx 7.2$ K the S/T and U_o/k_B are monotonic in the whole field regime. In contrast, for $T < T_o \approx 7.2$ K the respective quantities are non-monotonic. In addition, for magnetic fields $H > H''_{\text{smp}}(T)$ S/T and U_o/k_B present a monotonic behavior as a function of temperature, while for $H < H''_{\text{smp}}(T)$ they are non-monotonic on T . As a consequence for $H < H''_{\text{smp}}(T)$ S/T and U_o/k_B should present also extrema as a function of the temperature. In all cases the magnetic field was normal to the surface of the film $\mathbf{H} \parallel \mathbf{c}$.

may be described by a power-law $U_o \propto H^\nu$ with $\nu \approx -2$. This value is very high and probably inconsistent with the notion of plastic vortex creep. The differences between the relaxation process observed in Nb films and in high- T_c compounds as discussed above, may give additional evidence that the feature of the second peak effect is motivated by different physical mechanisms.

The relaxation measurements performed in temperatures $T > T_o \approx 7.2$ K showed different behavior. In Fig. 7 we present comparative data for S/T (upper panel) and U_o/k_B (lower panel) at $T = 2.5, 3.5, 4.3, 5$ K (below T_o) and at $T = 7.5, 8$ K (above T_o). In the upper panel we observe that the difference $S/T|_{\text{max}} - S/T|_{\text{min}}$ of the two extrema (as presented for example for the curve referring to $T = 3.5$ K) is higher at low temperatures. We may then conclude that as the characteristic temperature $T_o \approx 7.2$ K is approached, the extrema in S/T and U_o/k_B are suppressed and eventually disappear at T_o . Furthermore, we observe that both S/T and U_o/k_B

exhibit opposite dependence on T in the regimes below and above the "SMP" points. For $H > H''_{\text{smp}}(T)$ S/T (U_o/k_B) is an increasing (decreasing) function of temperature, while for $H < H''_{\text{smp}}(T)$ both S/T and U_o/k_B are non-monotonic as a function of temperature. This means that for $H < H''_{\text{smp}}(T)$ both S/T and U_o/k_B exhibit extrema not only as a function of field but also as a function of temperature. In contrast, in the temperature regime above T_o the behavior is completely different. As we see at $T = 7.5$ and 8 K both S/T and U_o/k_B present a conventional monotonic behavior as they approach the upper-critical field $H_{c2}(T)$.

CONCLUSIONS

In summary, we presented magnetic measurements as a function of field, temperature and time in films of the isotropic Nb superconductor prepared by annealing during the deposition. In measurements as a function of field we observed a "SMP" feature, while TMIs in the magnetic moment were observed either as a function of field, temperature and time. The three lines $H_{\text{fj}}(T)$, $H''_{\text{smp}}(T)$ and $H_{\text{fp}}(T)$ connect at a characteristic point $(T_o, H_o) \approx (7.2 \text{ K}, 80 \text{ Oe})$. Below $(T_1, H_1) \approx (6.2 \text{ K}, 1600 \text{ Oe})$ the line $H''_{\text{smp}}(T)$ changes slope. Pronounced TMIs are observed for $T_1 \approx 6.2 \text{ K} < T < T_o = 7.2 \text{ K}$, while below T_1 the magnetic curves are almost regular. Interestingly, in this low-temperature regime $T < T_1$, the first flux jump field preserves a constant value $H_{\text{fj}}(T < T_1) = 40 \text{ Oe}$. This is in strong disagreement to theoretical proposals for thin film, or even bulk samples and remains to be explained.

Relaxation measurements showed that below $T_o \approx 7.2$ K both the activation energy U_o and the normalized relaxation rate S present a non-monotonic behavior either as a function of temperature or the applied field. The observed extrema in U_o and S are located at the onset $H_{\text{on}}(T)$ or the peak points $H''_{\text{smp}}(T)$ of the "SMP". This is a noticeable difference comparing to high- T_c superconductors where the respective features are observed at a characteristic field $H^*(T)$ placed well below the SMP point. This could give additional evidence that the "SMP" observed in Nb films is of different origin than the respective feature observed in high- T_c compounds. Although we are not able to reveal the underlying mechanism that motivates the "SMP", our results indicate that the "SMP" in Nb films is accompanied by an anomalous relaxation of vortices. In contrast to high- T_c compounds, in Nb films this anomalous relaxation could even be the driving cause of the observed "SMP".

Although of different underlying physical mechanisms, the similarities of the presented phase diagram of vortex matter of our Nb disordered film with the phase diagrams observed in high- T_c compounds calls for further experimental and theoretical investigation.

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